93-213788/26 A96 805 D21 E19 PROC 91.12.11 PROCTER & GAMBLE CO *WO 9311754-A1

PROCTER & GAMBLE CO
91.12.11 91US-805432 (93.06.24) A61K 9/50, 7/16
Microcapsules for reducing oral bacteria and providing breath protection - comprise shell material and core compsn. contg. breath protection agent and antimicrobial e.g. quat. ammonium salt, copper salt, zinc salt or triclosan (Eng)
C93-094791 N(CA FI NO) R(AT BE CH DE DK ES FR GB GR IE IT LU MC
NL
PT
SE)
Addnl. Data: HUNTER M A, STAPLER J H
92.12.07 92WO-US10500

Microcapsules suitable for reducing oral bacteria and providing breath protection comprise a shell material suitable for use in the mouth and ingesting and a core compsn. comprising a breath protection agent antimicrobial selected from quat. ammonium salts, other cationic salts, copper salts, zinc salts, and/or triclosan, and an organic diluent.

ADVANTAGE

.

The microcapsules reduce oral bacteria and provide long lasting breath protection.

The microcapsules do not have the problems associated with microcapsules of the prior art, e.g. the wall of the microcapsules may develop imperfections and A(12-V4B, 12-W5) B(4-C3B, 5-A3A, 7-D4A, 10-A22, 12-A1, 12-L3, 12-M11E) D(8-B8) E(5-L3, 7-D4A, 10-A22)

cause loss of the contents prematurely, or the actives may not be easily solubilised in the materials usually present in the core.

PREFERRED MICROCAPSULES

The shell material is polyvinyl alcohol, gelatin, waves, gums or sugar candies.

The microcapsules are in the form of a sphere, oblong, disk, a puffed square, or a cylinder and the breath control agent is a quat. ammonium salt (e.g. cetyl pyridinium chloride) and/or domiphen bromide.

The microcapsules are 2-9mm in dia, and the shell wall thickness is 30 μ - 2 mm.

EXAMPLE

Microapsules were prepd. by mixing the following components (wt.%) of the core in one container and the components of them in another container: gelatin (12.578), sorbitol soln. (70% aq.; 2.046), saccharin (0.372), FD and C Blue#1 (0.002), FD and C yellow#5 (0.002) Captex 300 (a triglyceride; 72.140), flavour (12.750), cetyl pyridinium chloride (0.100) and domiphen bromide (0.010).

© 1993 Derwent Publications Ltd

WO9311754-A

Principal differences

- Microcognules > demenioner from > mm

- System reeds to be retained in the most in Whentarity to provide release

- Not applicable to Art application ladeling in this whenter y action.

```
3/39/1
DIALOG(R) File 345: Inpadoc/Fam. & Legal Stat
(c) 2000 EPO. All rts. reserv.
11233141
Basic Patent (No, Kind, Date): WO 9311754 A1 930624
                                                   <No. of Patents: 009>
Patent Family:
                                 Applic No
                 Kind Date
                                             Kind Date
    Patent No
    EP 616526
                 A1 940928
                                EP 93900886
                                              Α
                                                  921207
    FI 9402765
                                FI 942765
                                              Α
                                                  940610
                  Α
                      940610
                                                  940610
    FI 9402765
                 A0 940610
                                FI 942765
                                              Α
                      940610
                                NO 942170
                                              Δ
                                                  940610
    NO 9402170
                  Α
    NO 9402170
                                NO 942170
                                              Α
                                                  940610
                  A0
                      940610
    TR 27527
                      950607
                                TR 1174
                                              Α
                                                  921209
                  Α
                                US 3080
                                                  930111
    US 5286496
                  Α
                      940215
                                              Α
                                US 150663
    US 5382424
                  Α
                      950117
                                              Α
                                                  931110
                                WO 92US10500 A
                                                  921207
                  A1 930624
                                                           (BASIC)
    WO 9311754
Priority Data (No, Kind, Date):
    WO 92US10500 W 921207
    US 805432 A 911211
    US 805432 B2 911211
    US 150663 A 931110
    US 805432 B1 911211
PATENT FAMILY:
EUROPEAN PATENT OFFICE (EP)
  Patent (No, Kind, Date): EP 616526 A1 940928
    CETYLPYRIDINIUM CHLORIDE AND DOMIPHEN BROMIDE IN ORGANIC SOLVENT.
      (English; French; German)
    Patent Assignee: PROCTER & GAMBLE
                                        (US)
    Author (Inventor): HUNTER MARY ANN (US); STAPLER JUDITH HILL (US)
              (No, Kind, Date): WO 92US10500
                                                W
                                                    921207; US 805432 A
    Priority
      911211
    Applic (No, Kind, Date): EP 93900886 A
                                              921207
    Designated States: (National) AT; BE; CH; DE; DK; ES; FR; GB; GR; IE;
      IT; LI; LU; NL; PT; SE
    IPC: * A61K-009/50; A61K-007/16
    CA Abstract No: * 119(08)079866U; 120(16)200201Z
    Derwent WPI Acc No: * C 93-213788; C 94-057206
    Language of Document: English
EUROPEAN PATENT OFFICE (EP)
  Legal Status (No, Type, Date, Code, Text):
                                           PRIORITY (PATENT APPLICATION)
    EP 616526
                      911211 EP AA
                  Ρ
                               (PRIORITAET (PATENTANMELDUNG))
                              US 805432 A
                                             911211
                                            PCT-APPLICATION (PCT-ANMELDUNG)
    EP 616526
                  Ρ
                      921207
                              EP AA
                              WO 92US10500 W
                                                 921207
                                           EP-APPLICATION (EUROPAEISCHE
                       921207
    EP 616526
                  P
                              EP AE
                              ANMELDUNG)
                              EP 93900886 A
                                                921207
                       940928
                                           DESIGNATED CONTRACTING STATES IN
    EP 616526
                              EP AK
                              AN APPLICATION WITH SEARCH REPORT (IN EINER
                              ANMELDUNG BENANNTE VERTRAGSSTAATEN)
                              AT BE CH DE DK ES FR GB GR IE IT LI LU NL PT
                              SE
    EP 616526
                       940928
                              EP A1
                                            PUBLICATION OF APPLICATION WITH
                               SEARCH REPORT (VEROEFFENTLICHUNG DER
                               ANMELDUNG MIT RECHERCHENBERICHT)
                                           REQUEST FOR EXAMINATION FILED
                       940928 EP 17P
```

clare

EP 616526

Ρ

```
(PRUEFUNGSANTRAG GESTELLT)
                               940616
                P
                              EP 170
    EP 616526
                      950329
                                            FIRST EXAMINATION REPORT
                               (ERSTER PRUEFUNGSBESCHEID)
                               950214
                                            REFUSED (ZURUECKGEWIESEN)
    EP 616526
                  P
                      970730
                              EP 18R
                               970316
FINLAND (FI)
  Patent (No, Kind, Date): FI 9402765 A
                                         940610
    CETYLPYRIDINIUMKLORID OCH DOMIFENBROMID I ETT ORGANISKT LOESNINGSMEDEL
      (Swedish)
    Patent Assignee: PROCTER & GAMBLE (US)
Author (Inventor): HUNTER MARY ANN (US); STAPLER JUDITH HILL (US)
    Priority (No, Kind, Date): WO 92US10500 W
                                                921207; US 805432 A
      911211
    Applic (No, Kind, Date): FI 942765 A
                                            940610
    IPC: * A61K
    CA Abstract No: * 119(08)079866U; 120(16)200201Z
    Derwent WPI Acc No: * C 93-213788; C 94-057206
    Language of Document: Finnish; Swedish
  Patent (No, Kind, Date): FI 9402765 A0 940610
    CETYLPYRIDINIUMKLORID OCH DOMIFENBROMID I ETT ORGANISKT LOESNINGSMEDEL
      (Swedish)
    Patent Assignee: PROCTER & GAMBLE (US)
    Author (Inventor): HUNTER MARY ANN (US); STAPLER JUDITH HILL (US)
    Priority (No, Kind, Date): WO 92US10500 W
                                                921207; US 805432 A
      911211
    Applic (No, Kind, Date): FI 942765 A
                                            940610
    IPC: * A61K
    CA Abstract No: * 119(08)079866U; 120(16)200201Z
    Derwent WPI Acc No: * C 93-213788; C 94-057206
    Language of Document: Finnish; Swedish
FINLAND (FI)
  Legal Status (No, Type, Date, Code, Text):
    FI 942765
                A 980630 FI FD
                                            Application shelved (J tetty
                              sillens 4 kk)
NORWAY (NO)
                                           940610
  Patent (No, Kind, Date): NO 9402170 A
    Priority (No, Kind, Date): US 805432
                                                  911211; WO 92US10500 W
                                            Α
      921207
    Applic (No, Kind, Date): NO 942170 A
                                            940610
    IPC: * A61K-009/50; A61K-031/44; A61K-031/14; A61K-007/16
    CA Abstract No: * 119(08)079866U; 120(16)200201Z
   Derwent WPI Acc No: * C 93-213788; C 94-057206 Language of Document: Norwegian
                                      A0 940610
  Patent (No, Kind, Date): NO 9402170
    CETYLPYRIDINIUMKLORID OG DOMIFENBROMID I ORGANISK LOESNINGSMIDDEL
      (Norwegian)
   Patent Assignee: PROCTER & GAMBLE (US)
Author (Inventor): HUNTER MARY ANN (US); STAPLER JUDITH HILL
             (No, Kind, Date):
                                US 805432
    Priority
                                                  911211; WO 92US10500 W
                                            Α
      921207
   Applic (No, Kind, Date): NO 942170 A
                                            940610
    IPC: * A61K-009/50; A61K-031/44; A61K-031/14; A61K-007/16
    CA Abstract No: * 119(08)079866U; 120(16)200201Z
    Derwent WPI Acc No: * C 93-213788; C 94-057206
   Language of Document: Norwegian
TURKEY (TR)
  Patent (No, Kind, Date): TR 27527 A
    NEFES KORUYUCU MIKROKAPSUELLER (Turkish)
```

```
Patent Assignee: PROCTER & GAMBLE (US)
    Priority (No, Kind, Date): US 805432 A
                                             911211
    Applic (No, Kind, Date): TR 1174 A
                                         921209
    IPC: * A61K-007/16; A61K-007/00
    CA Abstract No: * 119(08)079866U; 120(16)200201Z
    Derwent WPI Acc No: * C 93-213788; C 94-057206
    Language of Document: Turkish
UNITED STATES OF AMERICA (US)
  Patent (No, Kind, Date): US 5286496 A
    BREATH PROTECTION MICROCAPSULES (English)
    Patent Assignee: PROCTER & GAMBLE (US)
    Author (Inventor): STAPLER JUDITH H (US); HUNTER MARY ANN (US)
    Priority (No, Kind, Date): US 805432 B2 911211
    Applic (No, Kind, Date): US 3080 A
                                         930111
    National Class: * 424490000; 424048000; 424049000; 424058000;
      424435000; 424440000; 424492000; 514948000; 514963000
    IPC: * A61K-009/50; A61K-009/16; A61K-009/68
    CA Abstract No: ; 120(16)200201Z
    Derwent WPI Acc No: ; C 94-057206
    Language of Document: English
  Patent (No, Kind, Date): US 5382424 A
                                          950117
    BREATH PROTECTION MICROCAPSULES (English)
    Patent Assignee: PROCTER & GAMBLE (US)
    Author (Inventor): STAPLER JUDITH H (US); HUNTER MARY A (US)
                                            931110; US 805432 B1 911211
    Priority (No, Kind, Date): US 150663 A
    Applic (No, Kind, Date): US 150663 A 931110
    National Class: * 424054000; 424049000; 424489000; 424492000
    IPC: * A61K-007/16; A61K-007/22; A61K-009/50
    CA Abstract No: * 119(08)079866U; 120(16)200201Z
    Derwent WPI Acc No: * C 93-213788; C 94-057206
Language of Document: English
UNITED STATES OF AMERICA (US)
  Legal Status (No, Type, Date, Code, Text):
                      911211 US AA
                                            PRIORITY
    US 5286496
                  Р
                              US 805432 B2 911211
                                           APPLICATION DATA (PATENT)
                      930111
                              US AE
    US 5286496
                  Ъ
                               (APPL. DATA (PATENT))
                              US 3080 A
                                            930111
                                            ASSIGNMENT OF ASSIGNOR'S
                              US AS02
    US 5286496
                  Ρ
                      930310
                              INTEREST
                              PROCTER & GAMBLE COMPANY, THE ATTN: CHIEF
                              PATENT COUNSEL IVORYDALE TECHNICAL CEN ;
                              STAPLER, JUDITH HILL: 19930111; HUNTER, MARY
                              ANN: 19930111
                                            PATENT
    US 5286496
                      940215
                              US A
                  Р
                                            EXPIRED DUE TO FAILURE TO PAY
    US 5286496
                  Р
                      980428
                              US FP
                              MAINTENANCE FEE
                              980218
                                            PRIORITY
    US 5382424
                  Ρ
                      911211
                              US AA
                              US 805432 B1 911211
                                           APPLICATION DATA (PATENT)
                      931110
                              US AE
    US 5382424
                  Ρ
                               (APPL. DATA (PATENT))
                              US 150663 A
                                              931110
                  Ρ
                      950117
                              US A
                                            PATENT
    US 5382424
                                            EXPIRED DUE TO FAILURE TO PAY
                      990330
    US 5382424
                  Р
                              US FP
                              MAINTENANCE FEE
                              990117
```

WORLD INTELLECTUAL PROPERTY ORGANIZATION, PCT (WO)
Patent (No,Kind,Date): WO 9311754 A1 930624
CETYLPYRIDINIUM CHLORIDE AND DOMIPHEN BROMIDE IN ORGANIC SOLVENT

PCT

(74) Agents: REED, David, T. et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45202 (US).

WORLD INTELLECTUAL PROPERTY ORGANIZATION



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT) (51) International Patent Classification 5: WO 93/11754 (11) International Publication Number: **A1** A61K 9/50, 7/16 24 June 1993 (24.06.93) (43) International Publication Date: PCT/US92/10500 (21) International Application Number: (81) Designated States: CA, FI, NO, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, (22) International Filing Date: 7 December 1992 (07.12.92) PT, SE). (30) Priority data: Published 11 December 1991 (11.12.91) US With international search report. 805,432 Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of (71) Applicant: THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH amendments. 45202 (US). (72) Inventors: HUNTER, Mary, Ann; 8368 Jadwin Street, Cincinnati, OH 45216 (US). STAPLER, Judith, Hill; 1084 Brown Road, Wilmington, OH 45177 (US).

(54) Title: CETYLPYRIDINIUM CHLORIDE AND DOMIPHEN BROMIDE IN ORGANIC SOLVENT

(57) Abstract

The present invention relates to oral compositions in the form of microcapsules which reduce oral bacteria and provide long lasting breath protection.

(English) Patent Assignee: PROCTER & GAMBLE (US)
Author (Inventor): HUNTER MARY ANN (US); STAPLER JUDITH HILL (US) Priority (No, Kind, Date): US 805432 A 911211 Applic (No, Kind, Date): WO 92US10500 A 921207 Designated States: (National) CA; FI; NO (Regional) AT; BE; CH; DE; DK; ES; FR; GB; GR; IE; IT; LU; MC; NL; PT; SE Filing Details: WO 130000 With international search report; Before expiration of time limit for amending the claims and to be republished in the event of the receipt of the amendments IPC: * A61K-009/50; A61K-007/16 CA Abstract No: ; 119(08)079866U Derwent WPI Acc No: ; C 93-213788 Language of Document: English WORLD INTELLECTUAL PROPERTY ORGANIZATION, PCT (WO) Legal Status (No, Type, Date, Code, Text): P 911211 WO AA PRIORITY (PATENT) WO 9311754 US 805432 A 911211 APPLICATION DATA (APPL. DATA) 921207 WO AE P WO 9311754 WO 92US10500 A 921207 DESIGNATED STATES CITED IN A WO 9311754 Ρ 930624 WO AK PUBLISHED APPLICATION WITH SEARCH REPORT

(DESIGNATED STATES CITED IN A PUBLISHED APPL. WITH SEARCH REPORT) CA FI NO DESIGNATED COUNTRIES FOR 930624 WO AL WO 9311754 P REGIONAL PATENTS CITED IN A PUBLISHED APPLICATION WITH SEARCH REPORT (DESIGNATED COUNTRIES FOR REGIONAL PATENTS CITED IN A PUBLISHED APPL. WITH SEARCH REPORT) AT BE CH DE DK ES FR GB GR IE IT LU MC NL PT SE PUBLICATION OF THE INTERNATIONAL Ρ 930624 WO A1 WO 9311754 APPLICATION WITH THE INTERNATIONAL SEARCH REPORT (PUB. OF THE INTERNATIONAL APPL. WITH THE INTERNATIONAL SEARCH REPORT) WO 9311754 Ρ 930902 WO DFPE REQUEST FOR PRELIMINARY EXAMINATION FILED PRIOR TO EXPIRATION OF 19TH MONTH FROM PRIORITY DATE

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

ATU BBB BBF BG BJ BR CCF CCH CCM CS CZ DE GS FI	Austria Australia Barbados Belgium Burkina Faso Bulgaria Benin Brazil Canada Central African Republic Congo Switzerland Côte d'Ivoire Cameroon Czechoslovakia Czech Republic Germany Denmark Spain Finland	FR GA GB GN GR HU IE IT JP KP KR LI LK LU MC MG MIL MN	France Gabon United Kingdom Guinea Greece Hungary Ireland Italy Japan Democratic People's Republic of Korea Republic of Korea Kazakhstan Liechtenstein Sri Lanka Luxembourg Munaco Madagascar Mali Mongolia	MR MW NL NO NZ PL PT RO RU SD SE SK SN SU TD TG UA US VN	Mauritania Malawi Netherlands Norway New Zealand Poland Portugal Romania Russian Federation Sudan Sweden Slovak Republic Senegal Soviet Union Chad Togo Ukraine United States of America Viet Nam
---	--	--	---	--	---

4

5

10

15

20

25

30

BROMIDE IN ORGANIC SOLVENT

TECHNICAL FIELD

The present invention relates to oral compositions in the form of microcapsules which reduce oral bacteria and provide long lasting breath protection.

BACKGROUND OF THE INVENTION

The use of breath control compositions such as breath mints, mouthwashes, chewing gums, etc. is widespread in most of the developed countries of the world. Another form which has been used are microcapsules containing a flavorant or other breath protection agent. These executions have acceptance due not only to their usefulness away from a place to expectorate mouthwashes but also due to the fact that they can be swallowed when the user does not need any more of the actives or doesn't want the microcapsule in the mouth any longer.

Although microcapsules have been used, there are problems associated with incorporating certain breath protection agents/antimicrobials into the core. Oftentimes the wall of the microcapsule may develop imperfections and cause loss of the contents prematurely. Additionally, the actives may not be easily solubilized in the materials usually present in the core.

The prior art discloses a variety of means for providing breath protection and reducing oral bacteria. Included among such means are sprays disclosed in <u>U.S. 3.431.208</u>, <u>March 4. 1969 to Bailey</u>. Particles containing an adhesive member are disclosed in <u>U.S. 3.911.099</u>, <u>October 7. 1975 to Den Foney et al</u>. Another form is a mouthwash concentrate in a unit dosage cup as disclosed in <u>U.S. 4.312.889</u>, <u>January 26</u>, <u>1982 to Melsheimer</u>. All of these references are incorporated herein by reference.

The present inventors have found that by incorporating the breath control/antimicrobial actives into the core of the microcapsule along with organic diluents, problems associated with other microcapsule executions can be avoided.

45

PCT/US92/10500

5

10

15

20

25

30

It is therefore an object of the present invention to provide improved microcapsules.

It is another object of the present invention to provide microcapsules which provide improved breath control and reduce oral bacteria.

It is still another object of the present invention to provide improved methods of providing breath control and reducing oral bacteria.

These and other objects will become more apparent from the detailed description which follows.

All percentages and ratios used herein are by weight unless otherwise specified. Additionally, all measurements are made at 25°C unless otherwise specified.

SUMMARY OF THE INVENTION

The present invention in one of its aspects relates to microcapsules which contain breath control actives/antimicrobials in the core of the microcapsule along with an organic diluent.

DETAILED DESCRIPTION OF THE INVENTION

The essential as well as optional components of the capsules of the present invention are described in the following paragraphs.

Capsule Shell Material:

The shell material of the microcapsules of the present invention can be any materials which are suitable for ingestion as well as retention in the oral cavity. Materials which are suitable include gelatin, polyvinyl alcohols, waxes, gums and sugar candy type materials used in cough drops and mints, for example.

The shell material is used to form any of a wide variety of shapes such as spheres, oblong shapes, disks, puffed squares and cylinders. The shell thickness is preferably in the range of about 30um to about 2mm, preferably from about 70um to about 110um. If the microcapsules are spherical, the particle diameter is generally in the range of from about 2mm to about 9mm, preferably from about 3mm to about 7mm.

Breath Control Agents/Antimicrobials Present in the Core:

The breath control agents used in the cores of the microcapsules include quaternary ammonium salts such as pyridinium salts (e.g., cetyl pyridinium chloride), domiphen bromide, other cationic materials such as chlorhexidine salts, zinc salts and copper salts. Other organic agents such as triclosan and other noncationic water insoluble agents are also useful herein. Such materials are disclosed in U.S. Patent 5,043,154, August 27, 1991, incorporated by reference herein.

10

15

20

25

30

5

These breath control/antimicrobial agents are used in an amount of from about 0.001% to about 2%, preferably from about 0.005% to about 1% of the total core contents.

Diluents for Use in Microcapsule Core:

The solubilizing agent for the breath control/antimicrobial agents used in the cores of the present microcapsules can be any of a number of materials. Preferred are oils such as corn, olive, rapeseed, sesame, peanut or sunflower. Other preferred materials are triglycerides such as Captex 300. These are used in an amount of from about 20% to about 80%, preferably from about 65% to about 70% of the total capsule weight.

Additional Agents Suitable for Use in the Core of Capsule:

The core of the microcapsules of this invention may contain any number of additional materials to provide additional efficacy and/or sensory perceptions. Such agents may include flavoring agents such as thymol, eucalyptol, menthol, methyl salicylate or witch hazel. These agents are used in an amount of from about .1% to about 25%, preferably from about 10% to about 15% of the total capsule weight.

In addition, a variety of sweetening agents such as sugars, corn syrups, saccharin or aspartame may also be included in the core. These agents are used in an amount of from about .1% to about 5%, preferably from about .35% to about .5% of the total capsule weight.

Method of Manufacture:

35

The capsules of the present invention can be made using a variety of techniques. One method is described after the following examples.

4

5

10

35

Industrial Applicability:

The capsules of the present invention are used by placing the capsules into the mouth and retaining them therein for a period sufficient to provide the desired effect.

The following examples further describe and demonstrate preferred embodiments within the scope of the present invention. The examples are given solely for the purposes of illustration and are not to be construed as illustrative of limitations of this invention. Many variations thereof are possible without departing from the invention's spirit and scope.

EXAMPLES 1-4

The following compositions/capsules are representative of the present invention.

	<u>Component</u>	<u>Weight %</u>			
15					
	Gelatin	12.578	12.328	12.578	17.578
	Sorbitol Solution (70% Aqueous)	2.046	2.05	2.046	2.046
	Saccharin	0.372	0.500	0.372	0.450
20	FD&C Blue #1	0.002	0.002	0.002	-
	FD&C Yellow #5	0.002	-	0.002	0.004
	Captex 300 ¹	72.140	70.00	71.925	66.142
	Flavor	12.750	15.00	12.75	13.500
25	Cetyl Pyridinium Chloride	0.100	-	-	-
	Domiphen Bromide	0.010	-	-	-
	Chlorhexidine	-	0.12	-	-
	ZnC12	•	-	0.025	-
	Sodium Lauryl Sulfate	-	-	0.300	-
30	Triclosan	-	-	-	0.28

1) Captex 300 is a triglyceride supplied by Capitol City Product, Columbus, Ohio.

The above compositions are prepared by mixing the components of the core in one container and the components of the shell in another container. The shell materials are heated to provide a fluid medium. The core and shell materials are then pumped

separately to a two-fluid nozzle submerged in an organic carrier medium. The capsules formed are allowed to cool and stiffen. They are then denatured and separated for further handling.

In the above compositions any of a wide variety of other shell materials, breath control agents, sweeteners as well as other components may be used in place of or in combination with the components listed above.

WHAT IS CLAIMED IS:

10

5

'n

15

20

25

30

35

نيو

- 1. Microcapsules suitable for reducing oral bacteria and providing breath protection comprising a shell material suitable for use in the mouth and ingesting and a core composition comprising a breath protection agent/antimicrobial selected from the group consisting of quaternary ammonium salts, other cationic salts, copper salts, zinc salts, triclosan and mixtures thereof and an organic diluent.
- 2. Microcapsules according to Claim 1 wherein the shell material is selected from the group consisting of polyvinyl alcohol, gelatin, waxes, gums and sugar candies.
- 3. Microcapsules according to either of Claims 1 or 2 wherein the microcapsule is in the form of a sphere, oblong, disk, a puffed square, or a cylinder and the breath control agent is a quaternary ammonium salt.
- 4. Microcapsules according to any of Claims 1-3 wherein the microcapsules are in the form of spheres.
- 5. Microcapsules according to Claim 4 wherein the microcapsules are from about 2mm to about 9mm in diameter and the shell wall thickness is from about 30um to about 2mm.
- 6. Microcapsules according to any of Claims 1-5 wherein the shell material is gelatin.
- 7. Use of a breath protection agent/antibacterial in the manufacture of microcapsules for reducing oral bacteria and breath odor in the mouth wherein the microcapsules comprise a shell material suitable for use in the mouth and ingesting and a core composition comprising a breath protection

3

agent/antimicrobial selected from the group consisting of quaternary ammonium salts, other cationic salts, copper salts, zinc salts, triclosan and mixtures thereof and an organic diluent.

- 8. A manufacture according to Claim 7 wherein the microcapsule shell is made of gelatin.
- 9. A manufacture according to either of Claims 7 or 8 wherein the breath control/antimicrobial active is selected from the group consisting of cetyl pyridinium chloride, domiphen bromide and mixtures thereof.
- 10. A method according to any of Claims 7-9 wherein the microcapsule is in the form of a sphere.

ternational application No.

PCT/US 92/10500

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: A61K 9/50, A61K 7/16
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: A61K

3

ð

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DIALOG: WPI, WPIL, CLAIMS, MEDLINE, EMBASE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO, A1, 8100205 (ARTHUR BARR), 5 February 1981 (05.02.81)	1-10
		
Υ .	WO, A1, 9015592 (PATRICK JOHN SHANAHAN), 27 December 1990 (27.12.90)	1-10
	·	
Y	EP, A1, 0485616 (SUNSTAR KABUSHIKI KAISHA), 20 May 1992 (20.05.92), see page 2 lines 1-40	1-10
	·	·
-		
I .		

X	Further documents are listed in the continuation of Box	C.	X See patent family annex.		
* *A*	Special categories of cited documents: document defining the general state of the art which is not considered	T	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention		
-E-	to be of particular relevance ertier document but published on or after the international filing date	"X"	considered povel or cannot be considered to involve an inventive		
L.	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	-y-	step when the document is taken alone document of particular relevance: the claimed invention cannot be		
-0-	document referring to an oral disclosure, use, exhibition or other		considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art		
P	document published prior to the international filing date but later than the priority date claimed	*&*	document member of the same patent family		
Dat	e of the actual completion of the international search	Date	of mailing of the international search report		
200	Marrah 1002		1 6. 04. 93		
Na	Name and mailing address of the ISA/		Authorized officer		
	European Patent Office, P.B. 5818 Patentiaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl. Fax: (+31-70) 340-3016	ANNE	ELI JÖNSSON -		

X See patent family annex.

INTERNATIONAL SEARCH REPORT

PCT/US 92/10500

	PCT/US 92/1	
C (Continu	ation). DOCUMENTS CONSIDERED TO BE RELEVANT	
Category*	and the relevant passages	Relevant to claim No.
Y	Dialog Information Services, World Patent Index - latest, File 351, Dialog accession no. 007957913, WPI Acc No: 89-223025/31, ((LIOY) LION CORP), "Compsn. for mouth, e.g. tooth paste - contg. copper cpds. selected from copper gluconate and its alkali metal salt and copper citrate, chlorhexidine gluconate etc.", JP 1153620, A, 890615, 8931	1-8,10
Y	US, A, 5043154 (ABDUL GAFFAR ET AL.), 27 August 1991 (27.08.91)	1-8,10
	·	
	• .	
1	ISA/210 (continuation of second sheet) (July 1992)	

INTERNATIONAL SEARCH REPORT Information on patent family members

26/02/93

International application No. PCT/US 92/10500

Patent document cited in search report		date	me	mber(s)	Publication date	
WO-A1-	8100205	05/02/81	AU-B- 531215		11/08/83	
.,			AU-A-	6223580	13/02/81	
			EP-A-	0022662	21/01/81	
			US-A-	4292028	29/09/81	
WO-A1-	9015592	27/12/90	NONE	.4		
EP-A1-	0485616	20/05/92	AU-A-	7893891	31/12/91	
			CN-A-	1057582	08/01/92	
			JP-A-	4036231	06/02/92	
			WO-A-	9118585	12/12/91	
US-A-	5043154	27/08/91	AT-B-	395109	25/09/92	
			AU-B-	615173	26/09/91	
			AU-B-	629742	08/10/92	
			AU-B-	630028	15/10/92	
	-		AU-A-	1017588	04/08/88	
			AU-A-	7423591	11/07/91	
			AU-A-	7423691	11/07/91	
			AU-A-	7423891	11/07/91	
			BE-A-	1001110	18/07/89	
			DE-A-	3802168	11/08/88	
			FR-A-	2610195	05/08/88 .	
			FR-A-	2640134	15/06/90	
			FR-A-	2647010	23/11/90	
			FR-A-	2647011	23/11/90	
			FR-A-	2647013	23/11/90	
			FR-A-	2669532	29/05/92	
			GB-A,B-	2200551	10/08/88	
			GB-A,B-	2230187	17/10/90 17/10/90	
			GB-A,B-	2230188 2230189	17/10/90	
			GB-A,B-		25/10/88	
			JP-A- NL-A-	63258404 8800206	16/08/88	
			SE-A-	8800299	31/07/88	
			US-A-	4894220	16/01/90	
			US-A-	5037635	06/08/91	
			US-A-	5037637	06/08/91	
			US-A-	5080887	14/01/92	
			US-A-	5156835	20/10/92	
	•		US-A-	5178851	12/01/93	
			US-A-	5180578	19/01/93	

This Page Blank (uspto)